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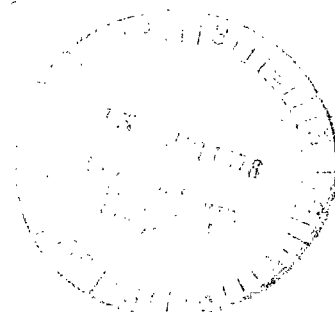
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# Effect of Oxygen and Nitrogen Interactions on Friction of Single-Crystal Silicon Carbide

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# Effect of Oxygen and Nitrogen Interactions on Friction of Single-Crystal Silicon Carbide

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# EFFECT OF OXYGEN AND NITROGEN INTERACTIONS ON FRICTION OF SINGLE-CRYSTAL SILICON CARBIDE

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## SUMMARY

An investigation was conducted to examine the nature of the interactions of oxygen and nitrogen with silicon carbide and of the friction behavior of silicon carbide sliding against itself and against titanium. Surface treatments examined included (1) exposure to gaseous oxygen and nitrogen (adsorption), (2) exposure to oxygen at high temperature (oxide film formation) and (3) bombardment with oxygen and nitrogen ions. Friction experiments were conducted in ultra high vacuum, and Auger emission spectroscopy was used to assess the presence of oxygen and nitrogen.

The results of the investigation indicate that the surfaces of silicon carbide with reacted-oxygen and ion-bombarded oxygen ions gives higher coefficients of friction than do argon-sputter-cleaned surfaces. The effects of oxygen in increasing the friction may be related to the relative chemical thermodynamic properties of silicon, carbon, and titanium to oxygen. The adsorbed films of oxygen, nitrogen, and mixed gases of oxygen and nitrogen on sputter-cleaned, oxygen-ion bombarded, and reacted-oxide surfaces generally results in the reduction of the friction of silicon carbide. The adsorption to silicon carbide is relatively weak. Nitrogen-ion bombardment results in the same coefficient of friction as that for the argon sputter cleaned surface.

## INTRODUCTION

The friction properties for a clean silicon carbide-metal contact system are related to the relative chemical activity of the metal for silicon or carbon. The more active the

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metal, the higher the coefficient of friction (ref. 1). The surface activity will be strongly affected by gas-interactions with the surface, for example, the adsorption of a gas, the chemical reaction of the surface with gas, and so on. These effects will be observed not only on metal surfaces, but also on nonmetal surfaces, such as silicon carbide surface.

At low pressures and room temperature, oxygen adsorbs on a clean silicon carbide surface to an equilibrium layer thickness of no more than two monolayers (ref. 2). The electrical and chemical properties of metal and semiconductor surfaces can be drastically affected by even small fractions of an adsorbed monolayer (ref. 2). Thus, it would be of interest to develop an understanding of tribophysical and chemical properties of silicon carbide surfaces treated with adsorbates such as oxygen and nitrogen. Further, in reference 3 it was shown that bombarding metal surfaces with oxygen ions produced beneficial tribological effects. Similar benefits might be achieved by bombarding non-metals with oxygen and other ions.

The objective of the present investigation was to examine the effects of the interactions of oxygen and nitrogen on the friction behavior of silicon carbide sliding against itself and against titanium. Clean surfaces were treated by (1) exposing them to oxygen and nitrogen (adsorption), (2) chemically reacting them with oxygen, and (3) bombarding them with oxygen and nitrogen ions. Friction experiments on the treated surfaces were conducted with the single-crystal silicon carbide (0001) surface in contact with single-crystal silicon carbide (0001) or with polycrystalline titanium surfaces. All experiments were conducted with loads up to 0.30 newton (30 g), at a sliding velocity of 3 millimeters per minute, and in a vacuum of  $10^{-8}$  pascal and 25° C. Auger emission spectroscopy analysis was used to monitor surface chemistry.

## MATERIALS

The single-crystal silicon carbide used in the experiments was a 99.9 percent pure compound of silicon and carbon and had a hexagonal close-packed crystal structure. The polycrystalline titanium was 99.97 percent pure and also had a hexagonal close-packed crystal structure. The contacting surfaces of the single-crystal silicon carbide and the polycrystalline titanium riders specimens were hemispherical with a radius of 0.79 millimeter (1/32 in.) and were mechanically polished with approximately 3-micrometer-diameter diamond powder and then 1-micrometer-diameter aluminum oxide ( $\text{Al}_2\text{O}_3$ ) powder. The titanium was then chemically polished; however, the friction properties of the chemically polished and mechanically polished titanium surfaces were not significantly different. The surfaces of the single-crystal silicon carbide disk specimens were mechanically polished with a 3-micrometer diamond powder and then with a 1-micro-

meter  $\text{Al}_2\text{O}_3$  powder. The surfaces of all specimens were rinsed with water and 200-proof ethyl alcohol.

## APPARATUS

The apparatus used in the investigation is capable of measuring adhesion, load, and friction. The apparatus was mounted in an ultra-high vacuum system, which also contains an Auger emission spectrometer (AES). The mechanism used for measuring adhesion, load, and friction is shown schematically in figure 1. A gimbal-mounted beam is projected into the vacuum chamber. On the beam are two flats, machined normal to each other with strain gages mounted on each one. The silicon carbide or titanium rider is mounted on the end of the beam. The load is applied to the beam normal to the disk, and that load is measured by the strain gage. Vertical sliding motion of the rider along the disk surface is accomplished by a motorized gimbal assembly. Under an applied load, the friction force is measured during vertical translation by the strain gage. Multiple wear tracks were generated on the disk surface by translating the beam containing the rider.

## PROCEDURE

The specimens were placed in the vacuum chamber, and the chamber was evacuated and baked out to a pressure of  $1.33 \times 10^{-8}$  pascal ( $10^{-10}$  torr). Then, argon gas was bled back into the vacuum chamber to 1.3 pascals. A 1000-volt, direct-current, negative potential was applied, with a positive electrode and specimens (both disk and rider) were argon-ion-sputter bombarded for 30 minutes. After the sputtering the vacuum chamber was reevacuated and Auger electron spectra (AES) of the disk surface was obtained to determine the degree of surface cleanliness. When the desired degree of cleanliness of the disk was achieved, friction experiments were conducted.

Loads of 0.1 to 0.3 newtons (10 to 30 g) were applied to the pin-disk contact by deflecting the beam (fig. 1). Both load and friction force were continuously monitored during a friction experiment. The sliding velocity was 3 millimeters per minute, and the total sliding distance was 2.5 millimeters. All friction experiments were conducted with the system evacuated to a pressure of  $10^{-8}$  pascal.

## RESULTS

### Silicon Carbide in Sliding Contact with Itself

Oxygen adsorption. - The crystal was in the as-received state after bake-out of the vacuum chamber. An Auger spectrum of the single-crystal silicon carbide surface, taken immediately after bake-out, shows the silicon peak height to be smaller than the carbon peak (fig. 2(a)). This condition may arise from the carbon contamination of silicon carbide (ref. 4). The Auger spectrum taken after the silicon carbide surface had been sputter cleaned clearly reveals the silicon and carbon peaks as well as a small oxygen peak. The residual oxygen may arise from the contamination of the bulk silicon carbide.

The silicon carbide was exposed to gaseous oxygen at a pressure of 1.3 pascals ( $10\text{ }\mu\text{m}$ ) and  $25^{\circ}\text{C}$  for 1 hour to insure surface saturation. If the oxygen peak height is compared with the silicon and carbon peaks in the resultant spectrum (fig. 2(c)) and with the corresponding peaks in figure 2(b), it is clear that more oxygen is present on the surface. The presence of the silicon peak (at 78 eV) indicates that the surface is covered with silicon oxide as well as with a simple adsorbed film of oxygen (ref. 5).

Sliding friction experiments were conducted with a single-crystal silicon carbide rider in contact with a single-crystal silicon carbide disk. The basal planes of both the rider and disk specimens were parallel to the sliding interface. The friction data, presented in figure 3, were obtained as a function of the number of passes over the same track for argon-sputter-cleaned surfaces and for surfaces containing adsorbed oxygen. The adsorbed film gave the lowest coefficient of friction after one to three passes of the rider. A marked increase in friction occurs when the number of passes is increased from 3 to 4, but beyond four passes the coefficients of friction are almost the same as those for sputter-cleaned silicon carbide. The marked difference in friction behavior is believed to be due to the breakdown of the adsorbed oxygen film. At a load of 0.10 newton (10 g), however, the breakdown of the adsorbed film did not occur for at least 20 passes. Thus, adsorbed oxygen films and oxides do reduce the friction of silicon carbide.

Oxygen-ion bombardment. - An argon-sputter-cleaned silicon carbide surface was next bombarded with oxygen ions at a 1000-volt potential, under a pressure of 1.3 pascals ( $10\text{ }\mu\text{m}$ ) at  $25^{\circ}\text{C}$  for 30 minutes. The spectrum of this surface (fig. 4(a)) has three characteristic peaks: silicon peaks at 68 and 82 electron volts (eV), a carbon peak at 272 eV, and an oxygen peak at 516 eV. The silicon peaks have two regions (I and II in the figure), which reflect the contribution to the valence band of silicon and oxygen bonding nonbonding molecular orbitals (ref. 5). The contribution from silicon-silicon bonds, which appeared in figure 2(a) at 92 eV, does not appear in this spectrum. Thus the oxygen-ion-bombarded silicon carbide surface seems to be covered by an  $\text{SiO}$  layer and,

above that, an  $\text{SiO}_2$  outer, or surface layer. The carbon peak, which was of the carbide type in figure 2(b), is nearly undetectable in figure 4(a). Friction data obtained for the oxygen-ion-bombarded silicon carbide surfaces (fig. 4(b)) indicate that the coefficients of friction for this surface are almost the same as or slightly higher than the coefficients of friction obtained for sputter-cleaned surfaces of silicon carbide (fig. 3).

A sputter-cleaned and then oxygen-ion-bombarded, silicon carbide surface was exposed to oxygen at a pressure of 1.3 pascals ( $10\ \mu\text{m}$ ) for 10 minutes. The Auger spectrum of this surface (fig. 5(a)) shows the silicon peak to be at 78 eV. The position of the peak reflects the contribution to the valence band of silicon and oxygen nonbonding molecular orbitals (ref. 5). The carbon peak is small, as it was in figure 4(a). The friction data for the silicon carbide surfaces in figure 5(b) show that at a load of 0.30 newton (30 g) the adsorbed film gives a fairly constant low coefficient of friction throughout the test without the breaking down.

Reacted oxide film. - Figure 6(a) is Auger spectra for silicon carbide which had been exposed to air at atmospheric pressure and  $700^\circ\text{C}$  for 10 minutes before sputter cleaning. After sputter cleaning (fig. 6(b)), the carbon peak is barely discernible in the spectrum, leaving an oxygen peak and a chemically shifted silicon-peak (at 82 eV), which indicates a layer of  $\text{SiO}_2$  on the silicon carbide surface. Figure 6(c) is the Auger spectrum of the silicon carbide surface after oxygen adsorption on reacted oxide film. If the oxygen peak height is compared with the silicon peak height and if these are compared with the corresponding peaks in figure 6(b), it is seen that a slightly higher oxygen peak is present on the surface.

The friction data, obtained for the surfaces with the reacted oxide of figure 6(b) and with an adsorbed film on the reacted oxide of figure 6(c), are presented in figure 6(d). The reacted oxide film gives a slightly higher coefficient of friction than the sputter-cleaned surface (fig. 3) or the oxygen-ion-bombarded surface (fig. 4(b)). The adsorbed oxygen film gives a lower coefficient of friction during the first three passes, but the same or slightly higher values beyond three. (Similar oxide-film behavior is shown in fig. 3.) The effects of oxygen on friction will be discussed in a later section (See section supplemental discussion.)

Nitrogen adsorption and ion bombardment. - Figure 7(a) is the Auger spectrum for a sputter-cleaned silicon carbide surface after exposure to gaseous nitrogen at 1.3 pascals ( $10\ \mu\text{m}$ ) and  $25^\circ\text{C}$  for 1 hour to insure saturation. The figure shows no nitrogen peak; however, if the silicon to carbon peak height ratio obtained from figure 7(a) is compared with that from figure 2(b), it can be seen that less silicon is present.

Next, sputter-cleaned silicon carbide surface was bombarded with nitrogen ions at a 1000-volt potential, at 1.3 pascals ( $10\ \mu\text{m}$ ) and  $25^\circ\text{C}$  for 30 minutes. Figure 7(b) is the Auger spectrum of this surface is the same as that of the sputter-cleaned silicon carbide surface (fig. 2(b)). Friction data obtained for the surfaces subjected to gaseous nitrogen and to nitrogen-ion bombardment are presented in figure 7(c). A reduction in

friction is noted for the adsorbed-nitrogen silicon carbide surface, but none for the nitrogen-ion bombarded surface. (Ion bombardment of the silicon carbide surface with a gas mixture of argon and nitrogen (in the ratio of 1 to 4) produced a nitrogen peak in the spectrum. The argon probably acted as a carrier gas which implanted the nitrogen ions in the silicon carbide. The friction behavior, however, was the same as that of ion bombarded surface. However, the absence of nitrogen peaks in figure 7 seem to arise from the electron induced desorption of nitrogen from the silicon carbide surface.)

### Silicon Carbide in Sliding Contact with Titanium

Oxygen adsorption and ion bombardment. - Both silicon carbide disk and titanium rider specimens were sputter cleaned at the conditions stated previously. The spectrum of silicon carbide is shown in figure 2(b). The Auger spectrum for the clean titanium surface shows titanium and a small oxygen peak, which arose from bulk titanium (ref. 3). One clean disk and rider set specimen was exposed to gaseous oxygen at 1.3 pascals ( $10\text{ }\mu\text{m}$ ) and  $25^{\circ}\text{C}$  for 1 hour; another set was bombarded with oxygen ions at 1000 volts, 1.3 pascals ( $10\text{ }\mu\text{m}$ ), and  $25^{\circ}\text{C}$  for 30 minutes. (See figs. 2(c) and 4(a) for spectra of the oxygen adsorbed and ion-bombarded surfaces of silicon carbide.) The oxygen to titanium peak height ratios indicated that the titanium surface contained slightly more oxygen after oxygen-ion bombardment than after oxygen exposure (ref. 3). With normal oxidation there is a layer of  $\text{TiO}$  between the metallic surface and the  $\text{TiO}_2$  layer.

The friction data obtained from these disk and rider sets are presented in figure 8 (the orientations of the single-crystal silicon carbide and the basal plane of the disk specimens are also shown). At one to three passes the adsorbed film gave the lowest coefficient of friction, but it apparently broke down after the third pass, as evidenced by the marked increase in friction. The oxygen-ion-bombarded disk and rider set had a slightly higher coefficient of friction, comparable with that of sputter-cleaned silicon carbide, but after repeated passes, its coefficient of friction generally decreased to an equilibrium value (0.56).

Reacted oxide film. - Figure 9 presents the friction data for a silicon carbide surface with a reacted-oxide film in sliding contact with a sputter-cleaned titanium surface. Figure 9 also presents the friction data for a silicon carbide surface with adsorbed oxygen on the reacted-oxide film in sliding contact with a titanium surface with an adsorbed oxygen film. The reacted oxide film gives a higher coefficient of friction than the argon-sputter-cleaned surface or the oxygen-ion-bombarded surface of silicon carbide. The coefficient of friction for the reacted-oxide film surface generally decreases with the number of passes to an equilibrium value (0.43), which depends on the nature of titanium transfer (ref. 1).



The adsorbed films on the reacted oxide of silicon carbide and titanium produces a low coefficient of friction under five passes. After eight passes a marked increase in friction occurred, arising from the breakdown of the adsorbed film.

Nitrogen adsorption and ion bombardment. - Figure 10 presents the friction data obtained for silicon carbide and titanium surfaces with adsorbed nitrogen or ion-bombarded nitrogen films. The adsorbed film reduced friction throughout the sliding without a breakdown. The nitrogen-ion-bombarded surface produced almost the same coefficient of friction as the argon-sputter-cleaned surface.

## DISCUSSION

### Oxygen and Nitrogen Interactions

Figures 11 and 12 summarize the coefficient of friction measured at a load of 0.30 newton (30 g), with a single pass of sliding for silicon carbide-to-silicon carbide interfaces and silicon carbide-to-titanium interfaces with reacted, ion-bombarded, and adsorbed oxygen or nitrogen films.

The reacted oxide and oxygen-ion-bombarded surfaces interact with the silicon carbide surfaces to produce two effects: (1) silicon carbide oxidizes and forms a protective oxide surface layer; and (2) the layer increases the coefficients of friction for both silicon carbide-to-silicon carbide and silicon carbide-to-titanium.

The effects of oxygen in increasing the friction is related to the relative chemical thermodynamic properties of silicon, carbon, and titanium to oxygen. Table I presents free energy of formation of silicides, oxides, carbides, and nitrides. The greater the degree of oxidation or oxygen implantation by ion bombardment, the more chemically active the surface (table I) and the higher the coefficient of friction (fig. 11). In such a situation oxygen will tend to chemically bond to the surface.

By contrast, adsorption of oxygen on argon-sputter-cleaned, oxygen-ion-bombarded, and reacted-oxygen surfaces generally decreases the coefficient of friction (figs. 12). When oxygen and nitrogen adsorb on the surface, the forces of attraction between the adsorbing gas and the surface of silicon carbide seem to be relatively weak.

The nitrogen bombardment gives essentially the same coefficients of friction as the sputter-cleaned surface. The adsorption of nitrogen or oxygen, however, reduces adhesion and friction.

## Mixed Gas Interactions

Figures 13 are the Auger spectra and friction data for silicon carbide surface after adsorption of a gas mixture of oxygen and nitrogen and in another set of experiments with ion bombardment of the surface with the gas mixture. The oxygen to nitrogen volume is in the ratio of 1 to 4. If the oxygen peak to carbon or silicon height ratios in figures 13(a) and (b) are compared with those in figures 2(c) and (d), it can be seen that less oxygen is present on the surface with adsorbed and ion-bombarded mixed gases than was observed on surfaces with pure oxygen. In figure 13(c) the friction behavior is similar to that for the surfaces with ion bombarded oxygen ions.

## CONCLUSIONS

As a result of the sliding friction experiments conducted in this investigation with single-crystal silicon carbide exposed to oxygen and nitrogen in various forms, the following conclusions are drawn:

1. Surfaces of silicon carbide which were reacted with oxygen and those ion-bombarded with oxygen give higher coefficients of friction than do argon-sputter-cleaned silicon carbide surfaces. The effect of oxygen in increasing the friction may be related to the relative chemical thermodynamic affinities of silicon, carbon, and titanium for oxygen.
2. The adsorbed films of oxygen, nitrogen, and mixed gases of oxygen and nitrogen on sputter-cleaned, oxygen-ion-bombarded, and oxygen-reacted surfaces generally reduce the friction of silicon carbide. The adsorption is, however, relatively weak.
3. Nitrogen ion bombardment results in the same coefficient of friction as that for the argon-sputter-cleaned surface.

Lewis Research Center,  
National Aeronautics and Space Administration,  
Cleveland, Ohio, April 12, 1978,  
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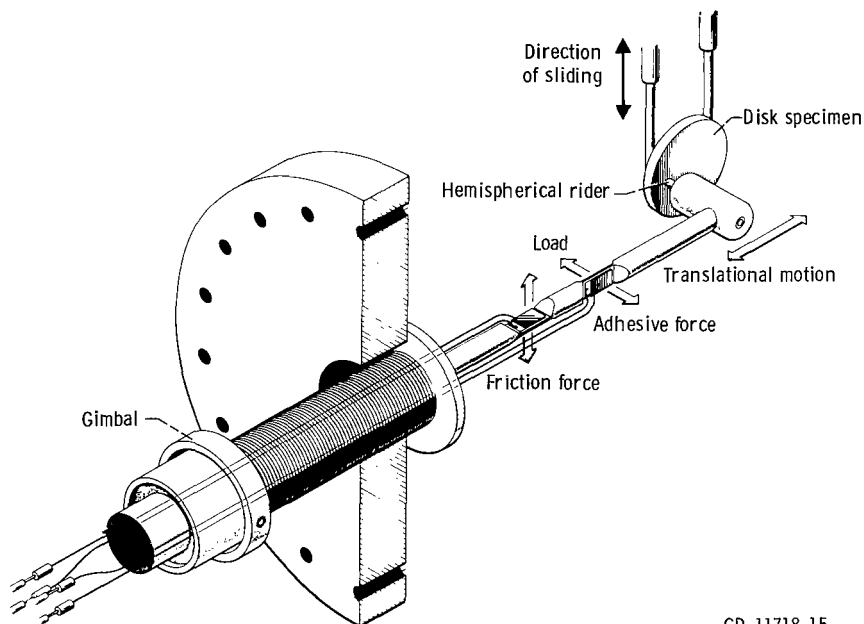
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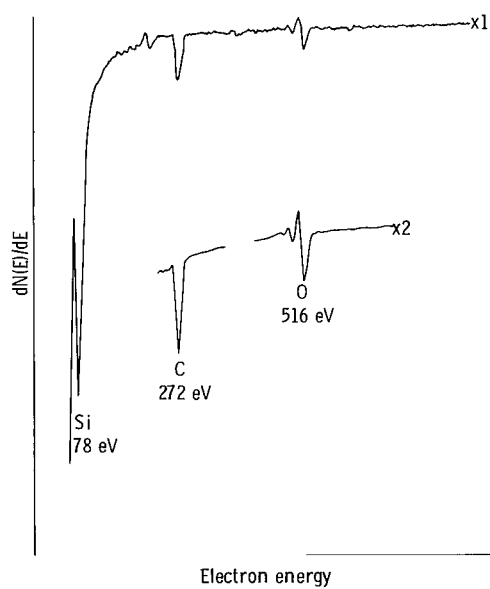
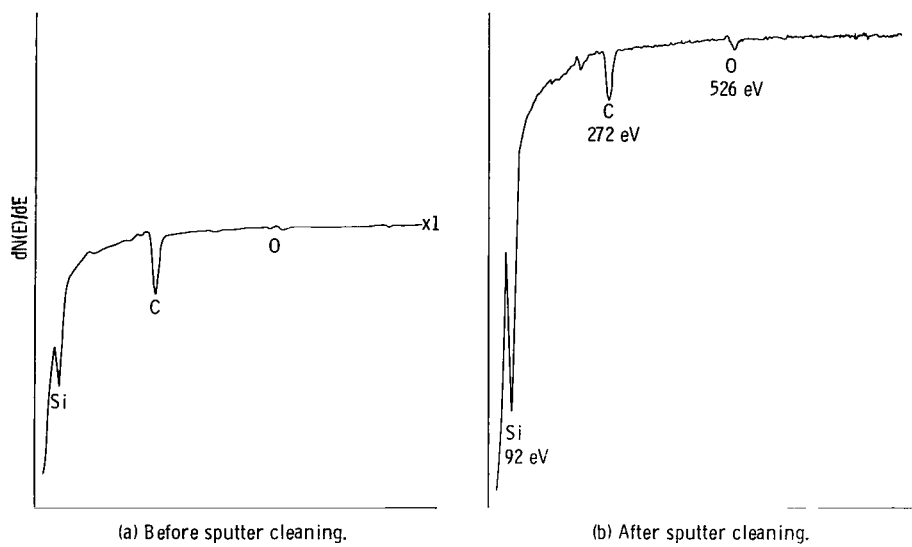
TABLE I. - VALUES OF FREE ENERGY OF  
FORMATION (REFS. 6 AND 7)

| Formula                        | Free energy of formation,<br>$\Delta F$ |                    |
|--------------------------------|-----------------------------------------|--------------------|
|                                | kcal/mole                               | J/mole             |
| SiC                            | -26.1                                   | $-109 \times 10^3$ |
| SiO                            | -32.77                                  | -137               |
| SiO <sub>2</sub>               | -192.4                                  | -805               |
| CO                             | -32.81                                  | -137               |
| CO <sub>2</sub>                | -94.26                                  | -394               |
| Si <sub>3</sub> N <sub>4</sub> | -154.7                                  | -647               |
| TiSi                           | -31.0                                   | -130               |
| TiC                            | -53                                     | -222               |
| TiO                            | -116.0                                  | -485               |
| TiO <sub>2</sub>               | -203.8                                  | -853               |
| TiN                            | -66.1                                   | -277               |



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Figure 1. - High-vacuum friction and wear apparatus.



(c) After exposure to gaseous oxygen (formation adsorbed oxygen film).

Figure 2. - Auger spectra of single-crystal silicon carbide (0001) surface.

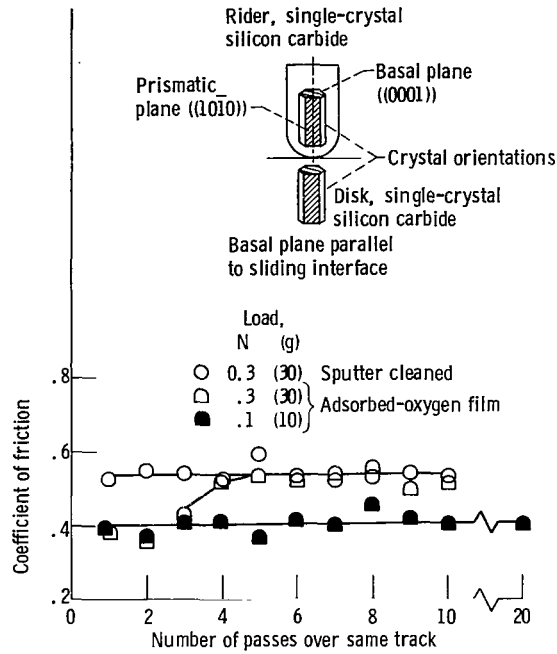


Figure 3. - Average coefficient of friction, obtained from maximum peak heights in friction trace, as function of number of passes of silicon carbide rider across silicon carbide (0001) disk. Oxygen adsorption conditions (disk and rider), 1.3 Pa (10  $\mu$ m) and 25 $^{\circ}$  C for 1 hour.

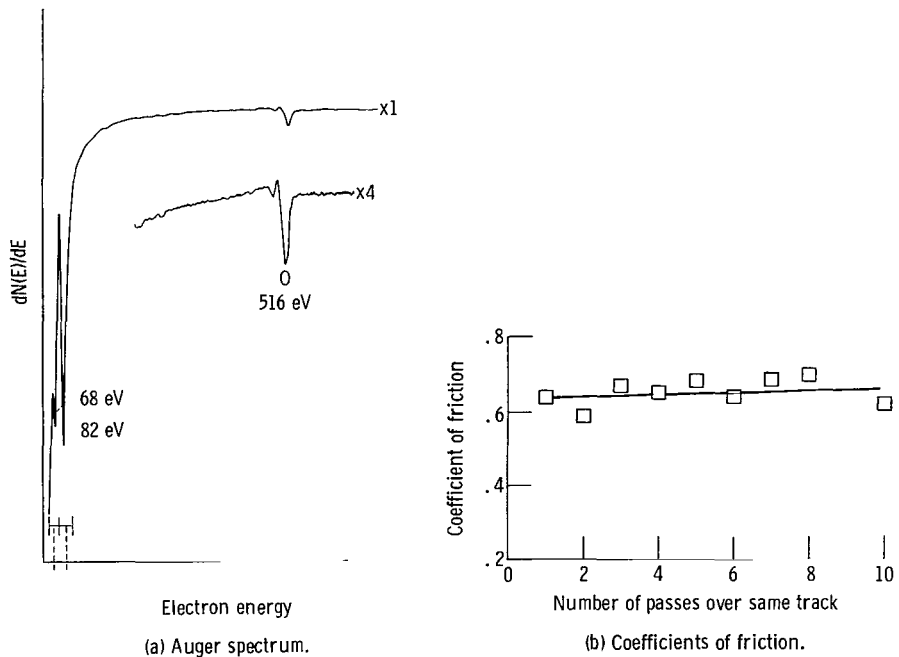


Figure 4. - Single-crystal silicon carbide (0001) surface with ion-bombarded oxygen film.

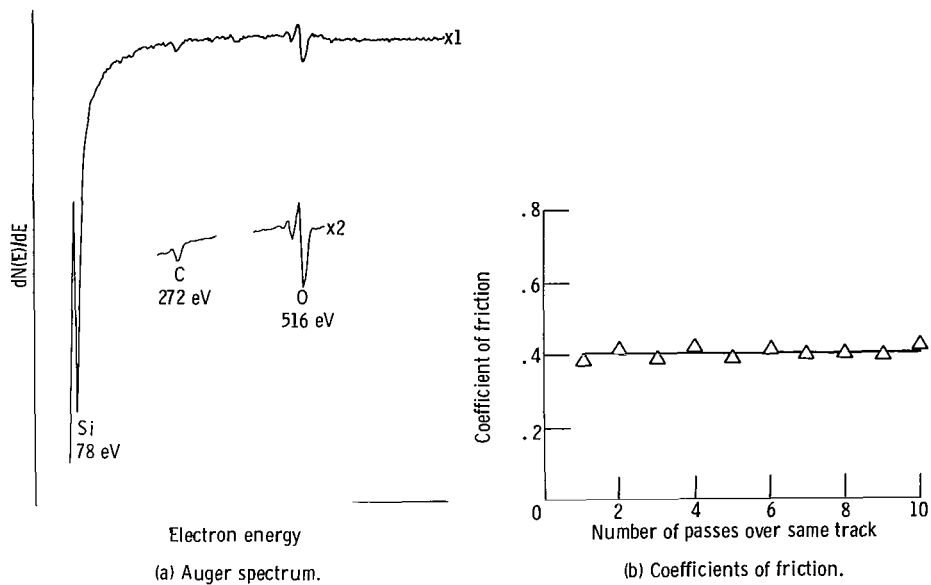


Figure 5. - Single-crystal silicon carbide (0001) surface with adsorbed oxygen film on an ion-bombarded oxygen film.

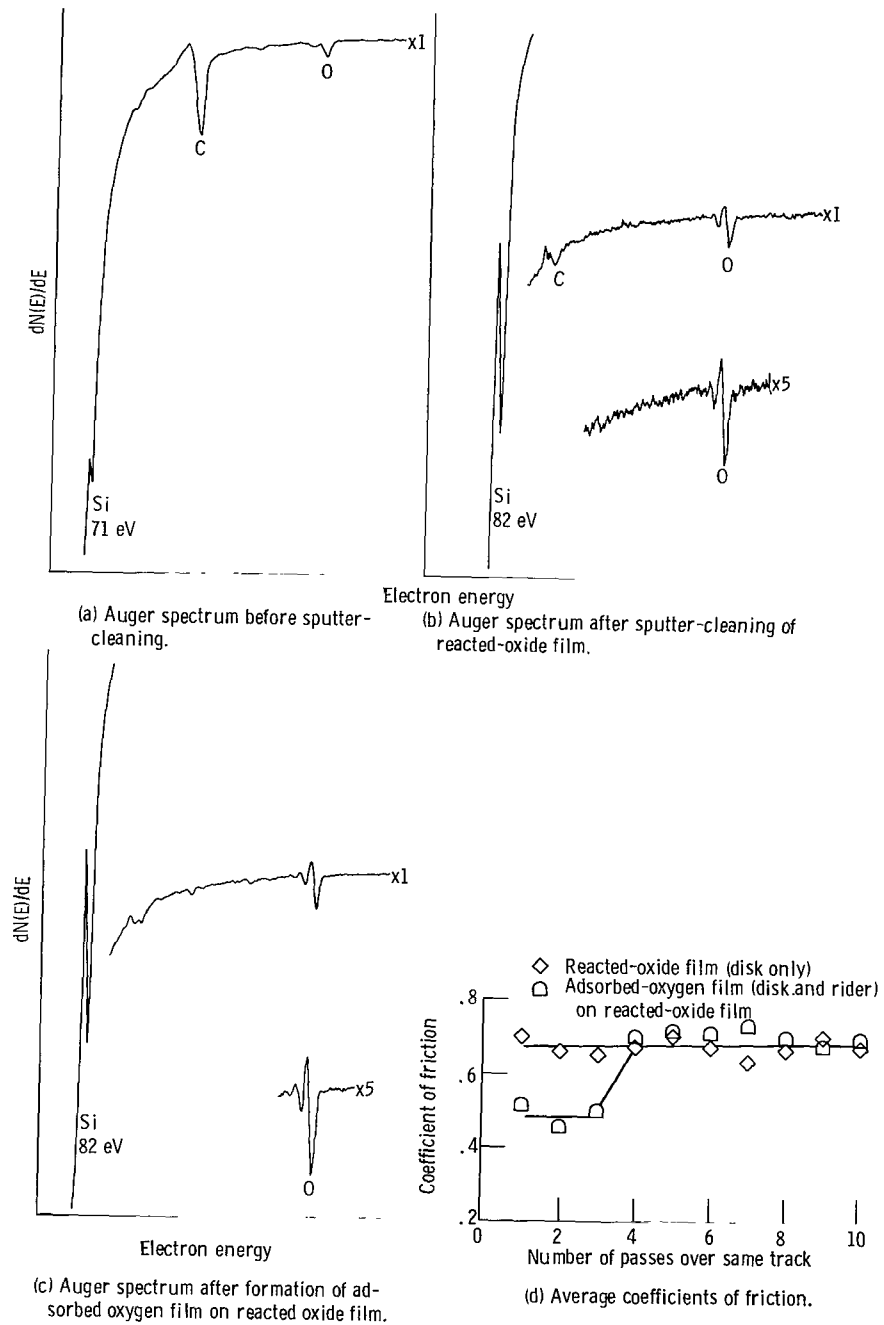
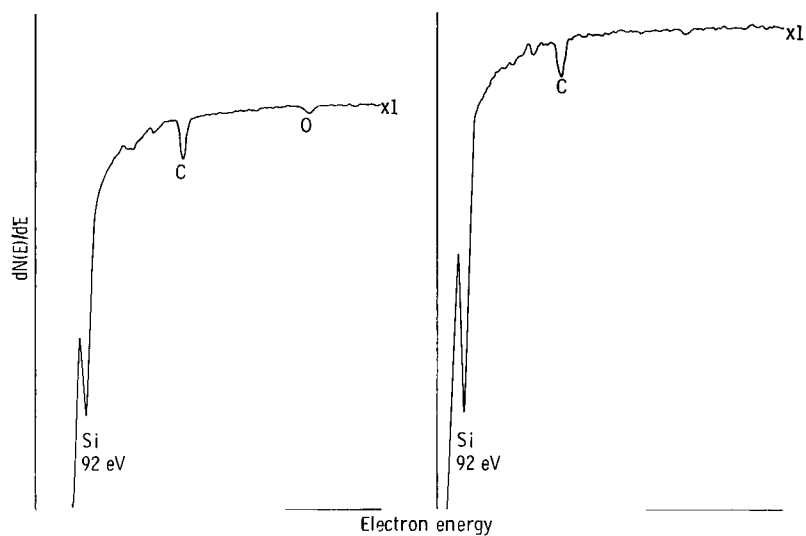


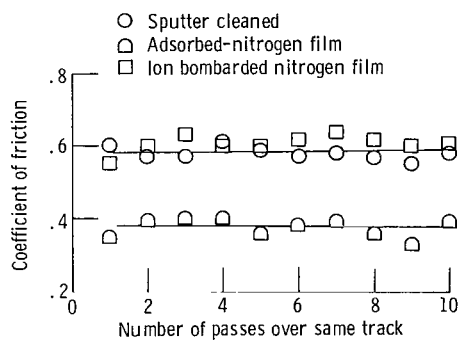
Figure 6. - Single-crystal silicon carbide (0001) surface with reacted-oxide film.





(a) Adsorbed nitrogen film.

(b) Ion-bombarded nitrogen film.



(c) Average coefficients of friction for silicon carbide sliding on silicon carbide.

Figure 7. - Nitrogen treated single-crystal silicon carbide (0001) surfaces

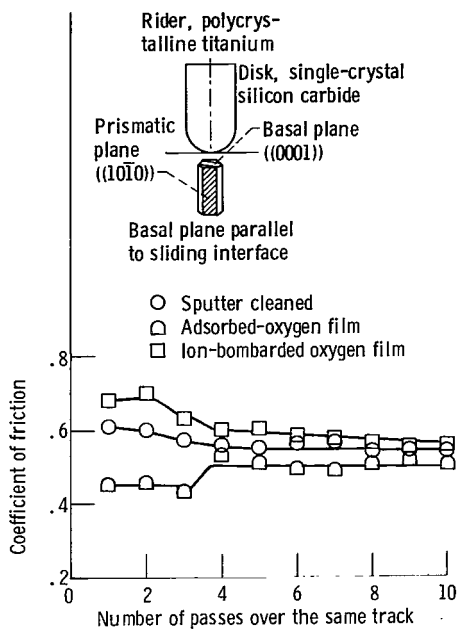


Figure 8. - Average coefficients of friction for titanium rider and silicon carbide disk sets (1) with adsorbed-oxygen film and (2) with ion-bombarded oxygen film.

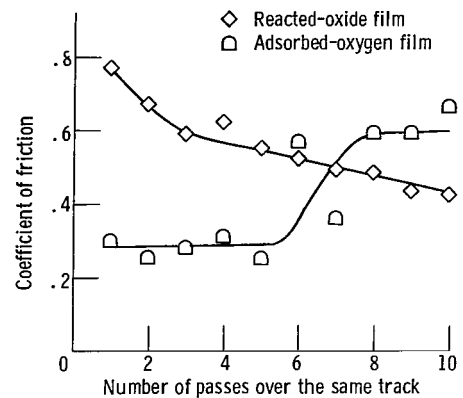


Figure 9. - Average coefficients of friction for silicon carbide with reacted-oxide film in sliding contact with sputter-cleaned titanium and for silicon carbide with adsorbed oxygen film on reacted-oxide film in sliding contact with titanium with adsorbed-oxygen.

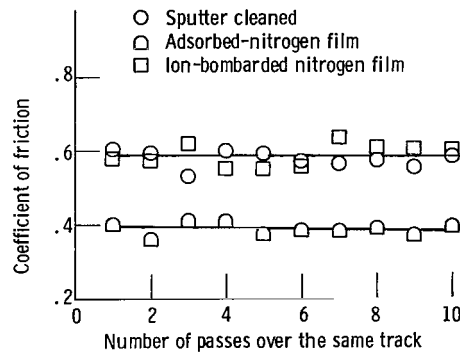
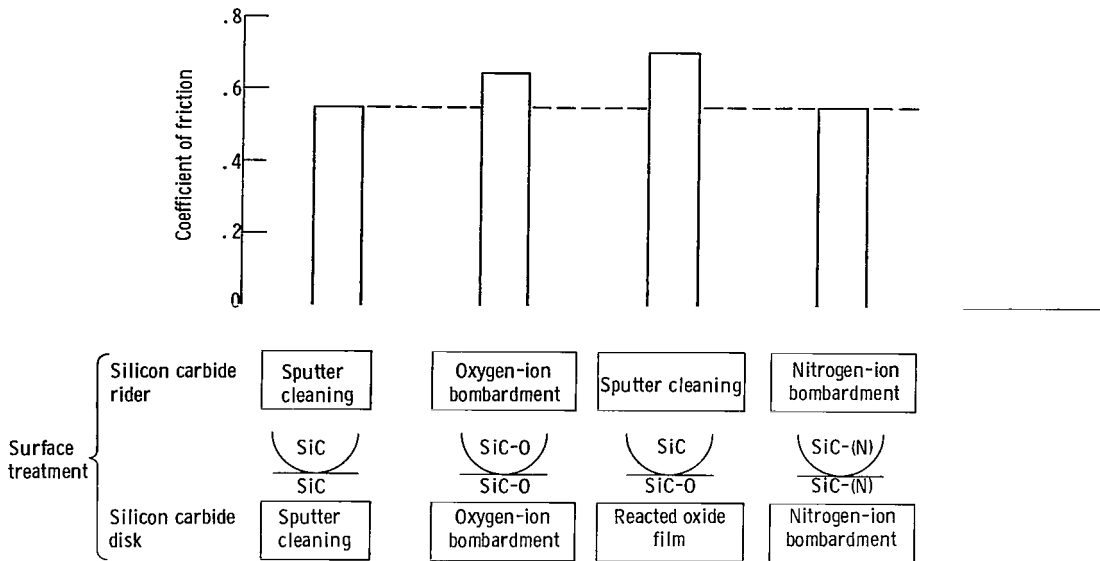
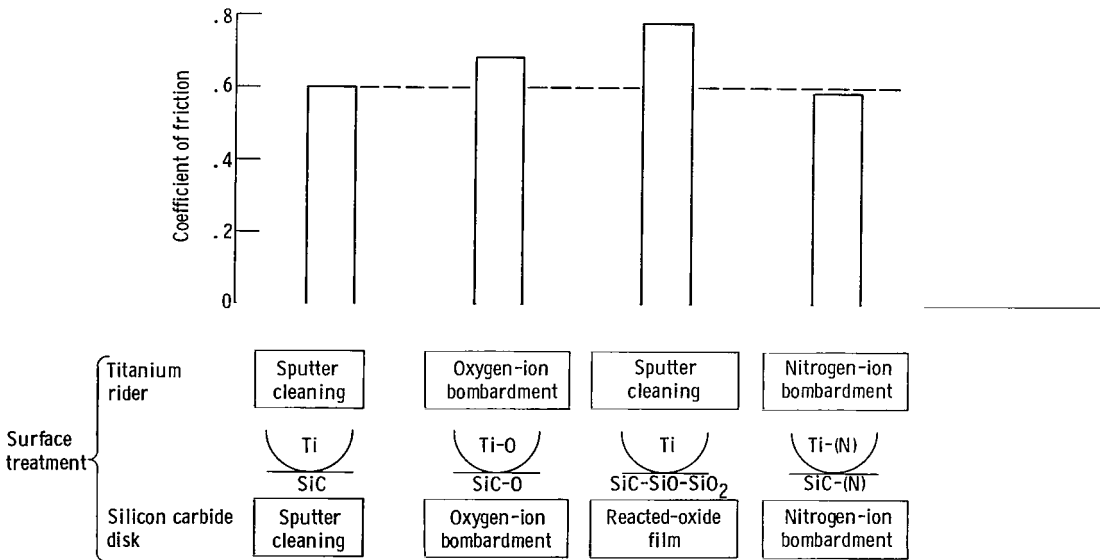


Figure 10. - Average coefficients of friction for titanium rider and silicon carbide disk (1) with adsorbed nitrogen film and (2) with ion-bombarded nitrogen film.

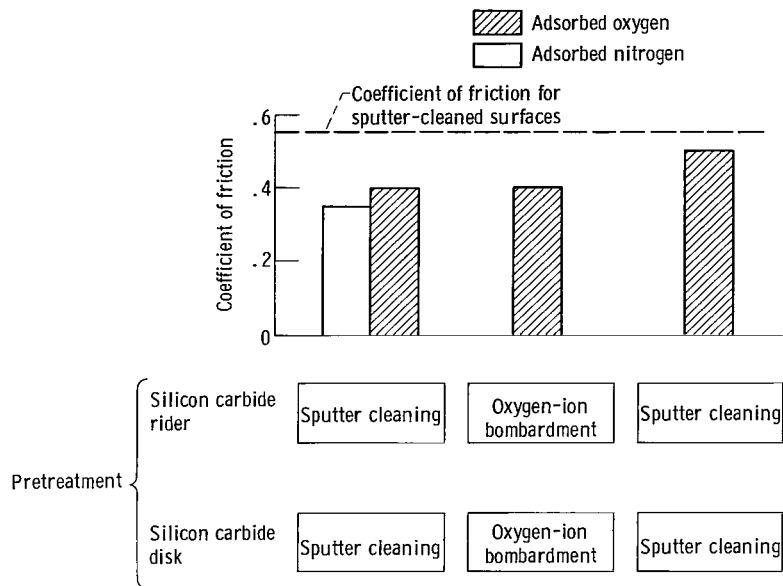


(a) Silicon carbide-to-silicon carbide contact.

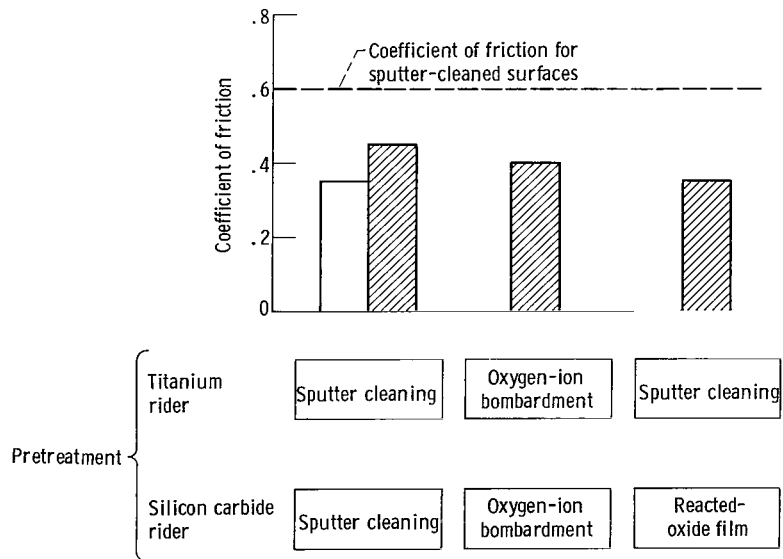


(b) Silicon carbide-to-titanium contact.

Figure 11. - Average coefficients of friction for silicon carbide-to-silicon carbide and silicon carbide-to-titanium contacts exposed to oxygen and nitrogen in various forms. Single pass sliding.

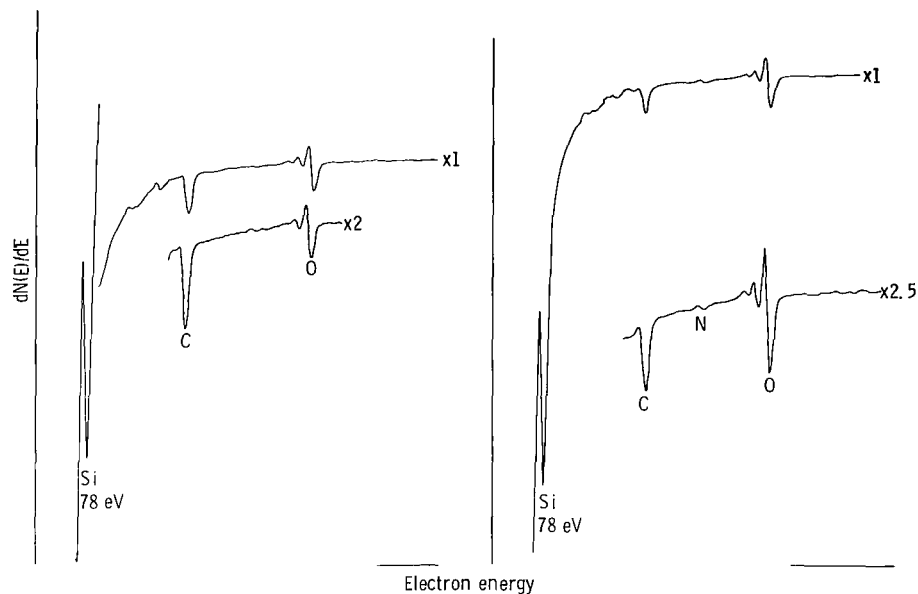


(a) Silicon carbide-to-silicon carbide contact.



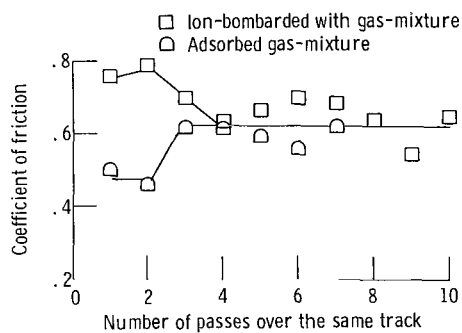
(b) Silicon carbide-to-titanium contact.

Figure 12. - Average coefficients of friction for silicon carbide-to-silicon carbide and silicon carbide-to-titanium specimens with adsorbed oxygen or nitrogen films after various pretreatments. Single pass sliding.



(a) Auger spectrum after formation of adsorbed gas mixture.

(b) Auger spectrum after mixed-gas ion bombardment.



(c) Coefficients of friction for surfaces with adsorbed and ion-bombarded mixed gas ions.

Figure 13. - Single-crystal silicon carbide (0001) surface with mixed-gas adsorption and ion bombardment.

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| 16. Abstract<br><p>Friction studies were conducted with single-crystal silicon carbide contacting silicon carbide and titanium after having been exposed to oxygen and nitrogen in various forms. After they had been sputter cleaned, the surfaces were (1) exposed to gaseous oxygen and nitrogen (adsorption), (2) ion bombarded with oxygen and nitrogen, or (3) reacted with oxygen (SiC only). Auger emission spectroscopy was used to determine the presence of oxygen and nitrogen. The results indicate that the surfaces of silicon carbide with reacted and ion-bombarded oxygen ions give higher coefficients of friction than do argon sputter-cleaned surfaces. The effects of oxygen on friction may be related to the relative chemical, thermodynamic properties of silicon, carbon, and titanium for oxygen. The adsorbed films of oxygen, nitrogen, and mixed gases of oxygen and nitrogen on sputter-cleaned, oxygen-ion bombarded, and oxygen-reacted surfaces generally reduce friction. Adsorption to silicon carbide is relatively weak.</p> |                                                                                    |                                                                 |
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